Final Report

Characterization and Eh/pH-Based Leaching Tests of Mercury-Containing Mining Wastes from the Sulfur Bank Mercury Mine, Lake County, California



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Notice

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Foreword

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory is the Agency's center for investigation of technological and management approaches for preventing and reducing risks from pollution that threatens human health and the environment. The focus of the Laboratory's research program is on methods and their cost-effectiveness for prevention and control of pollution to air, land, water, and subsurface resources; protection of water quality in public water systems; remediation of contaminated sites, sediments and ground water; prevention and control of indoor air pollution; and restoration of ecosystems. NRMRL collaborates with both public and private sector partners to foster technologies that reduce the cost of compliance and to anticipate emerging problems. NRMRL's research provides solutions to environmental problems by: developing and promoting technologies that protect and improve the environment; advancing scientific and engineering information to support regulatory and policy decisions; and providing the technical support and information transfer to ensure implementation of environmental regulations and strategies at the national, state, and community levels.

This publication has been produced as part of the Laboratory's strategic long-term research plan. It is published and made available by EPA's Office of Research and Development to assist the user community and to link researchers with their clients.

E. Timothy Oppelt, Director National Risk Management Research Laboratory

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ACRONYMS AND ABBREVIATIONS

ASTM American Society for Testing and Materials

BDL below detection limit

BEI backscattered electron image

ccm cubic centimeters per minute

CERCLA Comprehensive Environmental Response, Compensation, and Liability Act

CVAA cold vapor atomic absorption

EDS energy dispersive spectrophotometer

Eh redox equilibria (volt difference between platinum electrode and standard hydrogen

electrode)

Fe iron

 $Fe(NO_3)_3$ ferric nitrate

Hg mercury HgS cinnabar

H₂O₂ hydrogen peroxide H₂SO₄ sulfuric acid

ICDD International Centre for Diffraction Data

MDI Materials Data, Inc.

NaOH sodium hydroxide NPL National Priorities List

NRMRL National Risk Management Research Laboratory

NS not sampled

NTU nephelometric turbidity unit(s)

ORP oxidation-reduction potential

pH negative logarithm of the activity of aqueous H⁺

QAPP Quality Assurance Project Plan

RO reverse osmosis

SBMM Sulfur Bank Mercury Mine SEI secondary electron image SEM scanning electron microscope

SiO₂ silicon oxide, quartz

TCLP Toxicity Characteristic Leaching Procedure

TiO₂ anatase TO Task Order

TOL Task Order Leader TOM Task Order Manager UC UHP

University of Cincinnati ultrahigh purity United States Environmental Protection Agency U.S. EPA

XRD x-ray diffractometer

1.0 INTRODUCTION

Clear Lake in northern California has received inputs of mercury (Hg) mining wastes from the Sulfur Bank Mercury Mine (SBMM) (Figure 1-1). About 1.2 million tons of Hg-contaminated overburden and mine tailings were distributed over a 50-ha surface area due to mining operations from 1865 to 1957 (Gerlach et al., 2001). The SBMM includes an open, unlined mine pit, Herman Pit, which covers approximately 23 acres and is 750 feet upgradient of Clear Lake. Reynolds et al. (1997) analyzed water samples collected from Herman Pit and Clear Lake and reported the pH values at those locations as 3 and 8, respectively. The SBMM was placed on the Final National Priorities List (NPL) list in 1990. The site has been under investigation as a Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) site and has experienced some minor corrective actions. Clear Lake remains under a fish advisory due to the mercury contamination.



Figure 1-1. Location Map of Sulfur Bank Mercury Mine

Mercury in contaminated soils is a unique pollutant that requires innovative remediation solutions. Conventional stabilization/solidification treatments cannot effectively reduce the leachability of Hg (Conner, 1990). As part of the remediation effort at the SBMM site, the U.S. EPA is assisting in the development of treatment alternatives for waste material from the site. Waste materials consist of waste ore, waste rock, and roaster tailings. To support this work, leaching profiles of waste ore over a range of different pH and oxidation-reduction (Eh) conditions were performed. Chemical and biological processes affecting the mobility of metals may be initiated by altering the physicochemical environment (i.e., pH and Eh conditions). Important processes influencing the chemistry and availability of trace and toxic metals include (1) precipitation as insoluble sulfides under highly reduced conditions (Morel et al., 1974); (2) formation of discrete metal oxides and hydroxides of low solubility (Morel et al., 1974); (3) adsorption of colloidal hydrous oxides of iron and manganese, primarily in aerobic, neutral, or alkaline environments (Windom, 1973); and (4) complex formation with soluble and insoluble organic matter (Loganathan et al., 1977).

Each experiment has been designed to evaluate leachability of Hg from the waste materials under controlled conditions in order to assess conditions that may contribute to the destabilization of Hg in the waste ore.

1.1 Project Objective

The objective of this TO was to study a range of different pH and Eh values in order to evaluate the potential of SBMM waste ore to leach Hg. This study was conducted in accordance with the Quality Assurance Project Plan (QAPP) No. 63-Q1-3 (Battelle, 2001).

2.0 LABORATORY EXPERIMENT AND ANALYTICAL METHODS

Table 2-1 identifies both critical and noncritical measurements that were made during the course of this study. All analytical methods are described in QAPP No. 63-Q1-3 (Battelle, 2001) except the method for chloride analysis. (Chloride analysis was requested by the U.S. EPA TOM via e-mail on May 22, 2001.)

Table 2-1. Critical and Noncritical Measurements and Methods

Measurement	Method								
Critical									
Mercury	U.S. EPA SW-846 Method 7470A								
ORP	ORION® 96-78-00 Combination Redox Probe								
pН	U.S. EPA Method 9045C								
Noncritical									
Turbidity	Hach 2100N Turbidimeter								
Alkalinity/Acidity	U.S. EPA Method 310.1/305.1								
Chloride	U.S. EPA Method 407A								

ORP = oxidation-reduction potential.

2.1 Solid Material Preparation

The waste ore used in this study was obtained from the SBMM by the U.S. EPA. After receipt at Battelle, the waste material was homogenized, and then was ground for 8 hours and passed through American Society for Testing and Materials (ASTM)-approved No. 30 and No. 100 sieves to achieve particle sizes between 150 μ m and 600 μ m. The moisture from the samples was removed according to ASTM Method D2261-80.

2.2 Variable pH Leaching Procedure

The pH leaching procedure was based on University of Cincinnati's constant pH leaching procedure from QAPP No. 63-Q1-2 (UC, 1999). All experiments were conducted in accordance with the approved QAPP (QAPP ID No. 63-Q1-3) (Battelle, 2001).

To measure leachability at different pH values (2, 3, 4, 5, 6, 7, 8, 9, 10, 11, and 12), 25 g of dry solid sample were added in each 1-L bottle. Leachant at different pH values was prepared by adding nitric acid (0.1 N) or sodium hydroxide (0.1 N) in deionized reverse osmosis (RO) water. Duplicate samples were prepared for pH values 2, 5, 9, and 12. A solution to solid ratio of 20:1 was maintained in each of the bottles containing soil waste materials. The bottles were placed on a tumbler (Model 3740-12-BRE, Associated Design & Mfg. Co., VA) and equilibrated overnight. The pH was monitored frequently and adjusted as needed over the 24-hour time period. At the end of the reaction period, the pH of the leachant and the equilibrium pH of the solid-liquid suspension were recorded. ORPs of the leachate also were recorded after equilibration. Both pH and ORP were measured by a Corning pH/ion meter (Model 450). The ORP values were converted and are reported as Eh.

The above leaching procedure was followed in presence of ferric nitrate (Fe[NO₃]₃·9H₂O) (J.T. Baker, NJ) at four different pH values (3, 6, 9, and 11) to determine the effect of iron (Fe) on leaching of Hg. The amount of Fe(NO₃)₃·9H₂O added to each sample was based on the Hg concentration as observed from the previous set of experiments where no Fe was added. The amount of ferric nitrate was based on a final Fe concentration equal to the Hg concentration from the variable pH experiments (Table 2-2).

Table 2-2. Fe(III) Concentrations

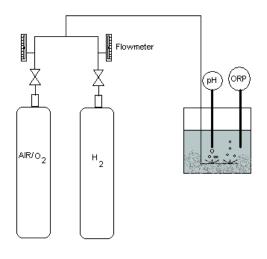
pН	Hg Concentration in Absence of Fe(III) (μg/L)	Fe(NO ₃) ₃ 9H ₂ O (mg)
3	15.3	0.055
6	728	2.633
9	1,938	7.009
11	4,020	14.540

2.3 Variable Eh Leaching Procedure

This section describes the experimental plan to determine the effect of Eh at two different pH values, on the mercury concentrations in the leachate. The pH of the leachant was maintained by adding suitable amounts of sulfuric acid (H_2SO_4) or sodium hydroxide (NaOH). The pH values selected by the U.S. EPA were 3.2 and 6.0. About 500 mL of the leachant was added to 25 g of prepared waste ore material, and the solution was readjusted to the desired pH. The Eh of the suspension then was varied by using one of the following three methods, without adding any chemicals: (1) purging the suspension with O_2 (to make the water aerobic); (2) purging the suspension with a mixture of O_2 and O_2 and O_3 purging the suspension with O_2 (to make the water anaerobic). About 1,670 O_2 and O_3 hydrogen peroxide (O_3) was added to achieve a higher Eh value of 0.63 V during only one set of experiments. All other experiments were conducted using different proportions of gas and gas flow control to establish target Eh values within the upper and lower Eh boundary conditions. To determine the effect of Fe(III), 0.01 g of Fe(O_3)3·9H2O was added in three samples during the variable Eh experiments (Table A-4). A schematic diagram and a photograph of the experimental setup are shown in Figure 2-1.

2.4 Filtration

After leaching, the suspended samples were filtered prior to Hg analysis. The suspension was passed through 0.7-µm Toxic Characteristic Leaching Procedure (TCLP) acid-treated low metal glass fiber filters (Whatman, UK) using a pressure filtration unit (Millipore Corp., MA) pressurized with ultrahigh purity





a. Schematic Diagram

b. Photograph

Figure 2-1. Variable Eh Experimental Setup

(UHP) nitrogen (see Figure 2-2). The filtrate from each sample was collected in a 500-mL bottle; a portion of the sample was acidified with nitric acid to obtain a pH less than 2 and stored inside the refrigerator at 4°C until analyzed for Hg. The remainder of the sample was sent to Wilson Environmental Laboratories (Columbus, OH) for either alkalinity/acidity analysis or chloride analysis.



Figure 2-2. Millipore Pressure Filtration Unit Pressurized with UHP Nitrogen

2.5 Analytical Procedures

The samples were prepared and analyzed according to U.S. EPA SW-846 Method 7470A: Mercury in Liquid Waste and Method 7471A: Mercury in Solid or Semisolid Waste by using a cold vapor atomic absorption (CVAA) spectrophotometry (Perkin Elmer 5100PC Atomic Absorption Spectrophotometer attached with Flow Injection Automated System), in which the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passed through a quartz cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) was measured at the 253.7-nm wavelength as a function of mercury concentration. The detection limit was $0.2~\mu g/L$. Total elemental analysis was conducted by acid digestion as per U.S. EPA Method 3050B of 1 g of solid sample to a final volume of 100 mL.

The turbidity of the filtrate was measured by using a Hach 2100N turbidimeter. Alkalinity and acidity were analyzed using U.S. EPA Methods 310.1 and 305.1, respectively. Chloride was analyzed using U.S. EPA Method 407A.

3.0 RESULTS AND DISCUSSION

The results of the experiments are discussed and analyzed below; analytical results are tabulated in Appendix A.

3.1 Characterization of Waste Materials

Selected physical and chemical properties of the SBMM waste ore material are given in Table 3-1. The material has an acidic pH and was moderately oxidizing. The homogenized and sieved solid samples were analyzed with an x-ray diffractometer (XRD), scanning electron microscope (SEM), and energy dispersive spectrophotometer (EDS). The XRD patterns (Figure 3-1) of the recovered crystalline phases were compared using organic and inorganic databases from the International Centre for Diffraction Data (ICDD) Powder Diffraction Database, and Materials Data, Inc. (MDI) Jade software for pattern treatment and search-match. In this analysis, the crystals anatase (synthetic TiO₂), cinnabar (HgS), and silicon oxide (SiO₂) were identified. The secondary electron images (SEIs), backscattered electron images (BEIs), and EDS elemental analyses are shown in Figures 3-2a through 3-2c. Secondary electron imaging shows topographic contrast, with highest resolution at low operating current. Backscattered electron imaging shows compositional contrasts, which are greatest at higher operating current.

3.2 Effect of Eh and pH Conditions

Eh and pH conditions are important influences on the mobility of Hg. Figure 3-3 illustrates different chemical forms of Hg under specific Eh and pH conditions. The data points (•) on the stability diagram show the different conditions achieved during the experiments. In general, metallic mercury is very insoluble in sediments over a wide pH range. Dissolved inorganic Hg combines with chloride up to a pH of 7. It exhibits a very high affinity for sulfide in mildly reducing environments, such as stream and lake sediments, forming insoluble mercuric sulfides (Wang and Driscoll, 1995). Dissolved Hg also sorbs strongly to sediment and suspended solids, including iron oxyhydroxides (Balogh et al., 1997). Gagnon and Fisher (1997) demonstrated that the binding strength of mercury to sediments is high and that less desorption occurs under acidic conditions.

Table 3-1. Selected Physical and Chemical Properties of SBMM Waste Ore

Properties	Analytical Result								
pН	3.2±0.10								
Eh	0.44±0.055 V								
Particle Size D	istribution								
Sand (>50 μm)	81%								
Silt (2-50 μm)	41%								
Clay (< 2 μm)	36%								
Carbon Content									
Organic	0.44% C								
Total	0.46% C								
Cation Exchange Capacity	6.5 mequiv/100g								
Elemental Analysis									
Mercury	206 μg/g								
Arsenic	3.9 µg/g								
Titanium	364.9 μg/g								
Lead	36.5 μg/g								
Sulfide	3285.4 μg/g								

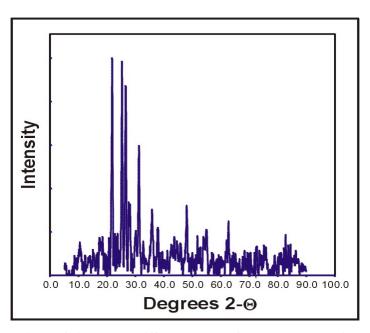
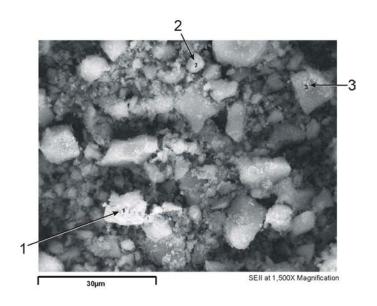


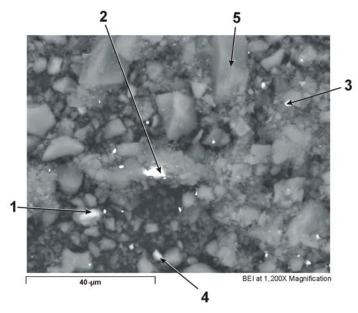
Figure 3-1. X-ray Diffractogram of the Waste Matrix



Spectrum	C	0	Al	Si	S	K	Ti	V	Fe	Cu	Hg	Total
1	6.50	13.14		2.24	9.20					0.78	68.13	100.00
2	18.90	53.74	0.31	12.07	0.19		14.27	0.28	0.24			100.00
3	24.80	47.84	3.43	22.10		1.19	0.38		0.27			100.00

All results in wt% and all elements are normalized.

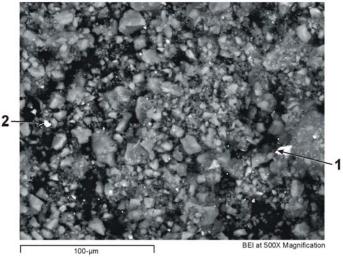
Figure 3-2a. SEI at 1500X, and EDS Analysis of Hg-Contaminated Waste Ore



Spectrum	C	0	Al	Si	S	K	Ti	Fe	Cu	Hg	Total
1	18.62	42.15	0.37	13.25			10.15	15.46			100.00
2	14.87	45.22	0.30	11.94	2.18	0.09	0.51		0.22	24.67	100.00
3	23.99	57.00	0.54	17.46	0.11	0.07	0.70	0.14	0.00		100.00
4	11.64	52.19	0.26	12.84	0.20		22.34	0.52			100.00
5	14.68	58.68	0.00	26.64							100.00

All results in wt% and all elements are normalized.

Figure 3-2b. BEI at 1200X, and EDS Analysis of Hg-Contaminated Waste Ore



Spectrum	C	0	Al	Si	S	K	Ca	Ti	Fe	Cu	Hg	Total
1		48.77	0.55	35.37	0.38	0.21	0.20	5.89	0.89	0.74	68.13	100.00
2	18.520	62.78	0.38	17.29	0.09	0.04		0.78	0.11	0.00		100.00

All results in wt% and all elements are normalized.

Figure 3-2c. BEI at 500X, and EDS Analysis of Hg-Contaminated Waste Ore

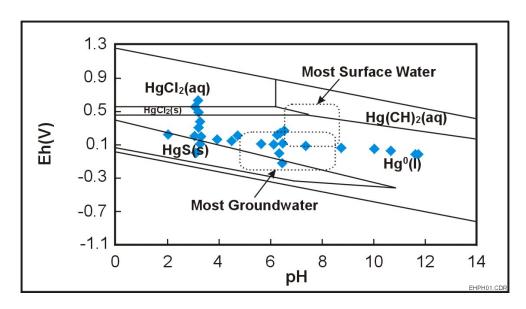


Figure 3-3. Leachability of Hg Conducted by TCLP Method at Different pH and Eh Conditions (modified after Davis et al., 1997; and surface and groundwater information obtained from Garrels and Christ, 1965). The data points (♦) on the stability diagram show the different conditions attended during the experiments.

The effect of pH on leaching of Hg from the waste materials is plotted in Figure 3-4. The Hg concentration increased as the equilibrium pH of the suspension increased to a pH value of 10.65. Thereafter, the Hg concentration decreased sharply. Acidity and alkalinity were measured for pH experiments and results are shown in Tables A-1 and A-2. Alkalinity concentrations were below detection level (BDL) (<1 mg/L) at low pH and increased as the sample pH increased. Acidity was highest at low pH and decreased BDL at pHs greater than 5. Eh of the suspension decreased as the pH increased. The Hg concentration, in presence of Fe(III), decreased significantly. The resulting Eh conditions, in presence and absence of Fe(III), during the experiments at different pH values are shown in Figure 3-5. The turbidity of the filtrate increased as the pH increased even though the same filters were used in all experiments (Figure 3-6).

The concentration of Hg in the leachate at variable Eh conditions is shown in Figure 3-7. During the experiments with variable Eh, the pH values were maintained 3.2 ± 0.08 and 6.4 ± 0.10 and both Eh and pH were monitored for about 24 hours. Each data point represents individual experiment and there were no replicates. Hg concentration, at pH 3.2, increased with increase in oxidation potential until the Eh value reached 0.2 V. Further oxidizing conditions reduced the leaching of Hg from the waste matrix. The concentration of Hg in the leachate increased sharply when H_2O_2 was added to attain a higher oxidation potential (Eh = 0.63 V). At pH 3.2, Fe(III) did not show any effect on leaching of Hg. The Hg concentration varied from 2.5 to 5.7 mg kg⁻¹ during the experiments, when the pH was maintained at 6.4. These experiments were conducted for about 24 hours. The rate of leaching of Hg at pH 3.2 in absence, and in presence of Fe(III) is shown in Figure 3-8. At the beginning of the experiment, Fe(III) releases Hg through oxidation. Burkstaller et al. (1975) reported leaching of Hg through oxidation of cinnabar in presence of Fe(III) in acid mine waters (pH <2.0). However, presence of Fe(III) reduces the rate of Hg leaching over a 24-hour period. The rates of dissolution of Hg from the waste ore at pH 3.2 are calculated as 1.02×10^{-7} s⁻¹ and 3.32×10^{-8} s⁻¹ in absence of Fe(III) and in presence of Fe(III), respectively. The change in chloride concentration as the Eh values change is shown in Figure 3-9.

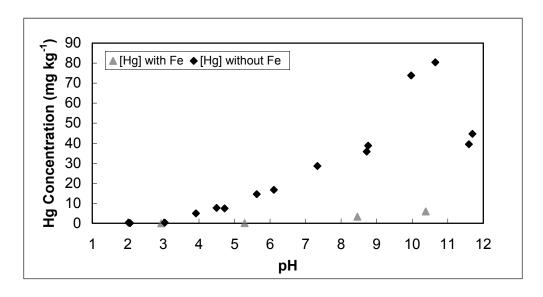


Figure 3-4. Concentration of Hg in the Leachate at Different pH Conditions

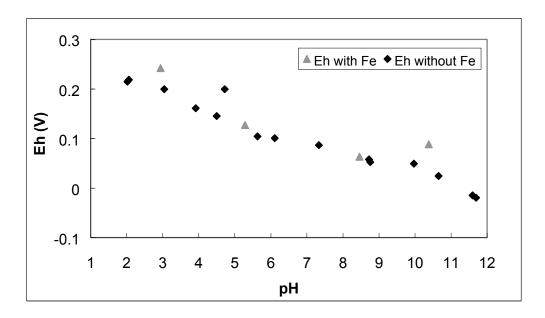


Figure 3-5. Eh Values at Variable pH Conditions Maintained During the Experiments

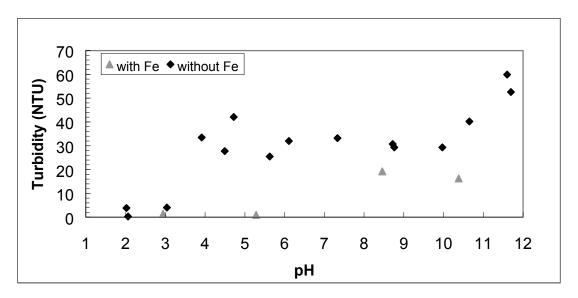


Figure 3-6. Turbidity of the Leachate at Different pH Conditions

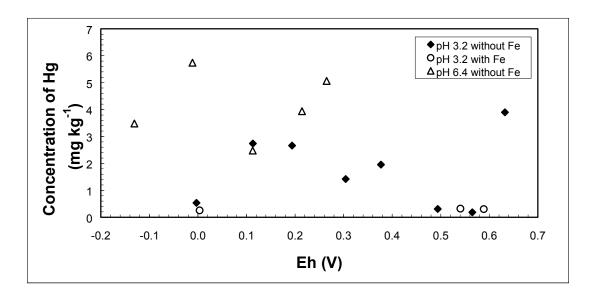


Figure 3-7. Concentration of Hg in the Leachate at Different Eh Conditions

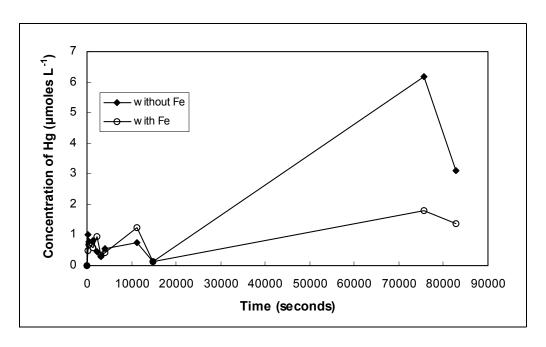


Figure 3-8. Leaching Rate of Hg With Fe (Eh 0.55 V) and Without Fe (Eh 0.5 V) at pH 3.2

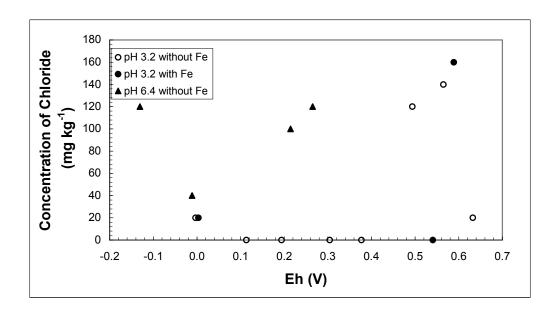


Figure 3-9. Concentration of Chloride in the Leachate at Different Eh Conditions

Alkaline and reduced conditions were found to enhance soluble levels of Hg. Wollast et al. (1975) reported that although the insoluble mercuric sulfide (cinnabar) will form in reducing environments, dissolved levels of Hg may increase in more strongly reducing conditions by conversion of the mercuric ion to the free metal form. Although the SBMM-water system studied for this report may differ from the Belgium River water described by Wollast et al. (1975), it is interesting to note that this study also detected higher levels of soluble Hg under strongly reducing conditions. The Eh-pH diagram (Figure 3-3) showed the comparatively soluble free metallic form to be stable compared to less soluble sulfide forms.

A composite leaching profile of Hg at different Eh and pH conditions is shown in Figure 3-10. Based on the leaching experiments, the concentration of Hg in the leachate (mg/L) was correlated to the different Eh (V) and pH values as follows:

Concentration of Hg in the leachate =
$$6.78 - 8.16 \times pH + 3.56 \times pH^2 - 0.7 \times pH^3 + 0.06 \times pH^4 - 0.002 \times pH^5 + 0.0004/Eh$$
.

The r^2 of the fitted equation was 0.96. The above correlation was obtained by using TableCurve $3D^{TM}$ (Jandel Scientific) software.

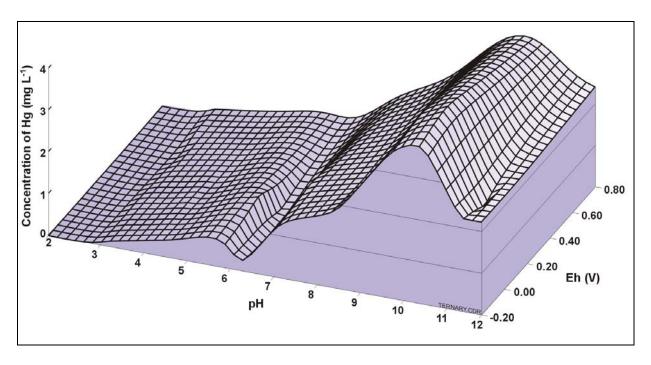


Figure 3-10. Composite Leaching Profile of Hg from the Waste Material at Different Eh and pH Conditions

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